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The Improvement of Properties Fly-Ash as Cementitious for Green Concrete

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Abstract: Fly-ash has a composition of silica that is dominant so that good used as cementitious. This research was conducted on the engineering materials of fly-ash from coal combustion waste at PLTU Tanjung Jati-B Jepara, Indonesia. Fly-ash which is synthesized by the method of reflux with through 5 M NaOH solution provided for 24 hours at a constant temperature of 100°C. Chemical testing done by X-Ray Diffraction (X-RD) and Scanning Electron Microscope (SEM). The result test will compared with fly-ash ordinary and cement material. Physical properties of fly-ash synthesized by the method of reflux has the potential of great absorption rate than conventional fly-ash. This is described with the inspection results of fineness fly-ash treatment retrieved by 13.4% and the value of consistency 37%. The reactivity of fly-ash with method of reflux also looks better silica compounds than fly-ash ordinary in where silica-oxide (SiO₂) of dominant 37,69% with amorphous phase reached 71,31% and round granules more smooth with size 6,425 µm. Changes the results of engineering materials, fly-ash with the reflux method can produce cementitious superior and environmentally friendly in the future.

Keywords: fly-ash, cementitious, reflux, reactivity.

1. Introduction

Fly ash is a by-product of Steam Power Plant (PLTU) that use coal as fuel, in the form of a light, smooth round granules, porous and not be pozzolanic. Chemically, the result of burning coal are volatile substances such as CO₂, SO₂, NO₂ and H₂O as well as non-volatile material in the form of coal-ash, known as basic gray (bottom-ash) and fly ash [7]. Fly-ash contains silica and aluminum, which reacts chemically with calcium hydroxide in the usual temperature forming compounds are cementitious [11]. But the use of fly-ash as a cementitious not optimal due to the dominant role of silica compounds in fly-ash is not reactive. The use of fly-ash by 30% as a filler instead generate strong press are better on the concrete than the use of fly-ash as a cementitious [5].

The purpose of this research is to know the physical properties and chemical repair of fly-ash as cementitious, as well as to know the influence of the reactivity of the material properties of the fly-ash which has under gone the process of synthesis. This research is expected to provide an alternative replacement of cement materials to obtain the green concrete in the construction of civil engineering.

The research geopolimer concrete [6] by doing a modification of fly-ash with variation of the concentration of NaOH solution start 1M, 2M, 3M produces the Si/Al ratio is higher than the composition of fly-ash before modification. Analysis of fly-ash through SEM test case morphological changes fly-ash before modification of spherical shape and smooth to rough on fly-ash after modification with 1M NaOH solution. Likewise for strength of geopolimer concrete achieved the highest strength and lowest porosity with fly-ash modification in 1 M NaOH solution.

Other studies [8] showed that fly-ash reflux into NaOH solution for 24 hours at a temperature of 90-100°C have an

increased surface area, pore radius and pore volume of fly-ash as well as change microstructure from amorphous to crystalline. While [9] has successfully to synthesis cation exchange from coal fly-ash with reflux method 4,5M NaOH solution at a temperature of 100°C for 2-48 hours.

1.1 Fly-ash

Fly-ash is the remains of burning coal, flowed from the combustion chamber by boiler in the form of a torrent of smoke, which has been used as an ingredient in concrete mixtures. Fly-ash themselves do not have the ability to bind such as cement. But with the presence of water and fine particles, silica-oxide conceived by fly-ash chemically reacts with calcium-hydroxide formed from cement hydration process and produce substances that have the ability to bind [4].

Fly-ash would be good enough to use as a binding material if its main constituent material having properties of a good reactivity as well as silicon dioxide (SiO₂), aluminium (Al₂O₃) and Ferrum oxide (Fe₂O₃). The oxide can react with lime which is released when the cement reacts with water. The use of fly ash as mixed mortar by 20-30% of the cement weight can to increase compressive strength of concrete. Physical properties of fly-ash has a high-fineness, round-shaped and non-porous [3].

Specifications fly ash as admixture concrete [1] are distinguished into three types:

- Types-N ; fly-ash kalsinasi results of pozzolan nature, e.g. the diatomite land, shore, tuft and pumice stone, usually processed through burning or not through the combustion process.
- Type-F ; fly-ash containing CaO less 10%, fly-ash resulting from burning the coal type anthracite at a temperature of approximately 1560°C. Fly-ash has pozzolan nature with

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the content of $(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3) > 70\%$

- Type-C ; fly-ash containing CaO at over 10%, and fly-ash resulting from the burning of coal or lignite with a carbon content of approximately 60% or sub-bituminous with the content of $(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3) > 50\%$

Table 1: The content of chemical fly-ash [1]

Komponen	Bituminous	Sub-bituminous	Lignite
SiO_2 (%)	20-60	40-60	15-45
Al_2O_3 (%)	5-35	20-30	20-25
Fe_2O_3 (%)	10-40	4-10	4-15
CaO (%)	1-12	5-30	15-40
Loi (%)	0-15	0-3	0-5

1.2 Cement

Cement is the most common construction material only in the workmanship of the concrete. Cement is produced by means of smoothing the clinker is composed of silicate calcium that is hidrolis with a cast as the admixture [2]

Table 2: The composition compound of cement [2]

Chemical Compound	%
Lime (CaO)	60-65
Silica (SiO_2)	17-25
Alumina (Al_2O_3)	3-8
Ferro (Fe_2O_3)	0.5-6
Magnesia (MgO)	0.5-4
Sulfur (SO_3)	1-2
Soda	0.5-1

2. Research on Methodology

2.1 Stage of Research

Research begins with physical test fly-ash Material and Concrete Laboratory of University Muhammadiyah of Malang, include: inspection of specific gravity, smoothness, consistency and setting-time of materials. The next stage is done the process of synthesis of fly-ash with the reflux method in Chemical Laboratory Muhammadiyah University of Malang by adding 5M NaOH solution during 24 hours at temperature 1000C. The next test is carried out a chemical composition and reactivity of fly-ash in the Sentral Laboratory of State University of Malang by using X-Ray Diffraction (X-RD) and Scanning Electron Microscopy (SEM). Stages of research shown in the figure-1.

2.2 Material and Instrument

The materials used on this research; fly-ash from coal waste PLTU Tanjung Jati-B Jepara, 5M NaOH (Sodium Hydroxide) solution. Research instrument used; X-Ray Diffraction (XRD), Scanning Electron Microscope, 1 set of instrument the synthesis of Reflux Method, Oven capacity of $100 \pm 50^\circ\text{C}$, 1 set of instrument gravity test.

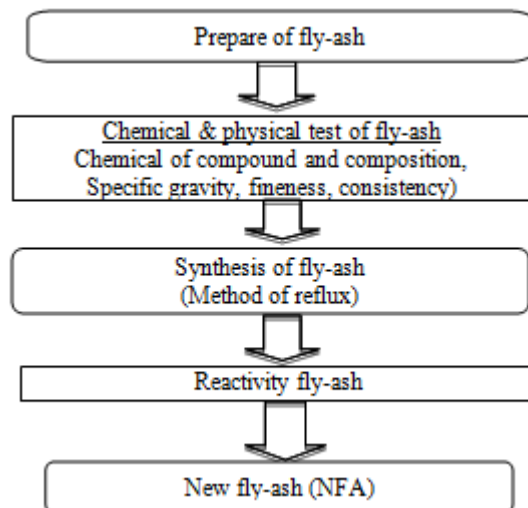


Figure 1: Stage of research

3. Result and Discussion

3.1 Characteristic of Fly-ash

Testing of the characteristics of fly-ash covers the fineness, gravity, and consistency. Materials tested consists of konvensional fly-ash, fly-ash has synthesis with reflux and cement as a comparison. The results of the physical test of materials listed in table-3.

Table-3 : Physical test of fly-ash and cement

Characteristic	Cement (PC)	Fly-ash (FA)	Fly-ash with reflux (FA-TR)
Fineness (%)	4.9	10.1	13.53
Specific Gravity (gr/cm^3)	3.17	2.625	2.55
Consistency (%)	27	28	37

The fineness of fly-ash has been to reflux will be more rough with a percentage of weight is stuck above the sieve No. 200 of 13.5%, while for konvensional fly-ash and cement fineness meet the requirements according to [1] as shown in Figure 2 (a). Does not satisfy the terms of fineness because hardened fly-ash due to synthesis process result NaOH concentration that is too dilute. The grain of fly-ash mutually bound during the process of synthesis takes place, so in dry condition resulting fly-ash feels more rugged compared to konvensional fly-ash. The reaction between cement and water starts from the surface of the cement grains, the increasing of the surface area caused faster happened cement hydration process. This means that the fine-grained cement will generate heat faster than hydration of cement with a grain of the more rough. In general, fine-grained cement improving cohesion on fresh concrete and can also reduce bleeding, but adds to a tendency of concrete to shrink more and simplify of crack shrinkage.

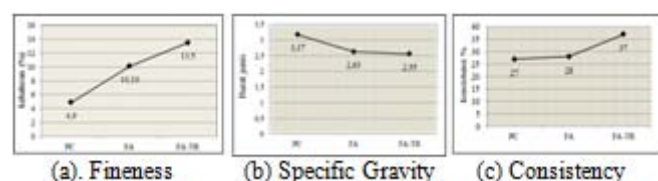


Figure 2: Result physical test of material

From Figure 2 (b) to see that the cement has a higher specific

gravity than conventional fly-ash and fly ash with treatment of reflux. Specific gravity of conventional fly ash and fly ash with treatment of reflux did not differ significantly although it has gone through the process of synthesis. Specific gravity of binder material (including of fly-ash) will affect specific gravity of the concrete it self. The use of fly ash as cementitious in the manufacture of concrete is expected to affect a concrete becomes lightweight. Based on Figure 2 (c) can be seen that the fly-ash has the consistency of treatment higher than cement and conventional fly-ash. This is based on the nature of these two different materials in the ability to absorb water. Fly ash is able to absorb more water in a cement to compare because fly ash due have amorphous structure higher than a lot of cement is dominated by crystalline structure. The amount of water mixed in the concrete will also affect the value of the cement water ratio (c/w) where the moisture content that is too large will cause segregation where water in the mixture will rise to the top of the concrete mix during the process of compaction is done. This condition is also referred to bleeding. When fly ash of reflux result was used as cementitious overall in the concrete mixture, then the water needed in the manufacture of concrete became much more. This is will resulted in concrete quality decreases as it contains too much water, but because the used fly-ash as a cementitious then it would just reduce the heat of hydration of cement in mix-concrete

3.2 Chemical Compound of Fly-ash

Results of inspection of chemical compounds with the test X-RD as shown in Table-4 explain there four dominant compound in cement materials are SiO_2 , Fe_2O_3 , Ca_3SiO_5 , and CaFe_3O_5 , with the most dominant Ca_3SiO_5 compounds (C_3S) formed from the reaction of silica and calcium which is the most important ingredient in cement. Silica oxide (SiO_2) is an important to compound binder in cement. It along with calcium oxide (CaO) formed a paste binder and the hydration reaction when mixed with water. For fly ash have four conventional constituent compounds namely, silica dioxide (SiO_2) is a form of quartz, calcium oxide (CaO), iron oxide/hematite (Fe_2O_3) and mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$).

The dominant compounds are SiO_2 (49,42%) and $\text{Al}_6\text{Si}_2\text{O}_{13}$ (29,27%). To mullite is a compound formed from the reaction of silica dioxide and alumina which both these compounds play an active role in shaping the bonds on fly-ash. This is due to both the most reactive compounds along with iron oxide (Fe_2O_3) is contained in fly-ash. Then there is also calcium oxide (CaO) is less dominant than with cement, low calcium can slow down the process of hydration and weaken the strength of the concrete.

Table-4 : The composition of compound material

Compound	Composition of compound (%)		
	Cement PC	Fly-ash FA	Fly-ash with reflux FA-TR
SiO_2 – Quartz	11,79	49.42	37,69
CaO – Calcium	-	10.03	-
Fe_2O_3 – Hematite	1,69	11.28	12,63
$\text{Al}_6\text{Si}_2\text{O}_{13}$ - Mullite	-	29.27	23,37
Ca_3SiO_5	83,7	-	-
CaFe_3O_5	2,83	-	26,63

Based on Table-4 composition of compound silica dioxide (SiO_2) on the fly-ash has decreased after the process of reflux, this decline is not caused by a missing element, but because a portion of active silica compound bound to be CaFe_3O_5 . In addition the process of reflux on fly-ash compound CaO will change into CaFe_3O_5 . This is due to undergo a consolidation with CaO 's compound iron oxide (Fe_2O_3) resulting in the compound to be changed. It can be seen on the results of the test compound, where CaFe_3O_5 has increased to a high of 26,63%. An increase has also occurred on the iron-oxide (Fe_2O_3) of 12,63%

Process for the synthesis of reflux change in total bond pure compounds in fly-ash, this is because the level of concentration sodium-hydroxide (NaOH) are quite high and the properties of alkali silica NaOH is a strong base. Chemical structure changing on the fly-ash after going through the process of synthesis, base chemical bonds, compound composition and basic structure of fly ash. This is confirmed by the results of previous studies [8] where by the process of with the method of synthesis of reflux with a solution of NaOH for 7 hours at a temperature of approximately 1000C, will result in a grains pozzolan have an increased surface area, pore volume and pore radius and modify the structure of amorphous became crystalline fly-ash.

3.3 Reactivity of Fly-ash

Examination of the level of reactivity of a material can be seen from the percentage of amorphous structure and its crystal structure. The analysis is done by reading the chart results of the integration of total output from X-Ray Diffraction (X-RD) devices. Table-6 describes the output area amorphous phase and crystal of the materials tested.

Table-5 : The composition of amorphous and crystalline structures

Specimen	Phase of area		Crystalline %	Amorphous %
	Amorphous	Crystalline		
Cement	299.58	1069.04	78.11	21.89
Fly-ash	1337.19	718.08	34.94	65.06
Fly-ash with reflux	1601.04	644.37	28.69	71.31

The reactivity of materials is very much dependent upon the amount of amorphous structure on a material. Amorphous form more easily experienced than dissolving crystalline form. This is due to crystal form is the form that is compact and has a more difficult ties penetrated the nucleophile molecules wide so not Si and Al having dissolving. Fly-ash PLTU Suralaya has a relatively amorphous phase more [10].

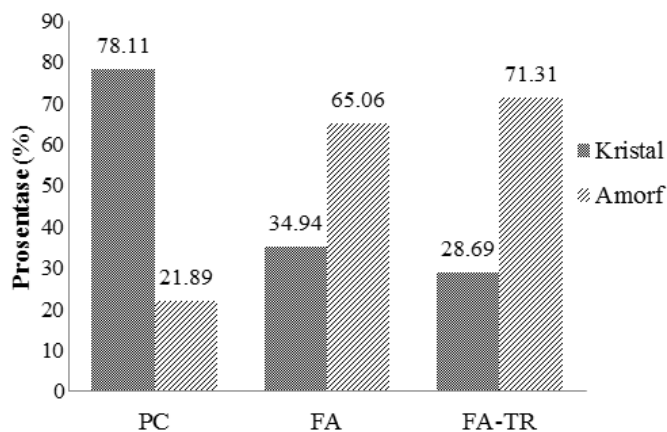


Figure 4: Composition of amorphous and crystalline structures.

Based on Figure-4 obtained different results between the ingredients of cement and fly ash, which cement has silica (SiO_2) compound 11,79% of which are amorphous. While the results of the integration of the cement material obtained diffraction of fly-ash has an amorphous phase of 21,89%. For conventional fly-ash amorphous nature has a structure more dominant of 65,06% with SiO_2 compounds of 49,42%. Influence of synthesis process of reflux provided materials of fly-ash will improve the properties of amorphous phase become 71,31% with SiO_2 compounds of 37,69%. This shows the nature of reactivity of fly-ash to be increased by the presence of the process of the synthesis of reflux. Silica plays a major role in adding properties reactivity materials even though a little percentage in those conditions (e.g. in cement simply 11,79% and fly-ash with reflux 37,69%) was able to activate the content calcium in the cement and fly-ash to bind and form the adhesive material to concrete.

Increasing silica reactivity on material properties of fly-ash can improve the dhesive paste as a result of fly-ash's reaction with water to the concrete constituent materials other (sand and coarse aggregate), but the reduction of compounds of CaO to be CaFe_3O_5 will make the process of hydration is become longer. So by changing the structure of silica is amorphous silica can be tied back-lime hydration reaction remains free so that the reaction can be more perfect, including material fly-ash used as cementitious part will generated material with adhesive properties better.

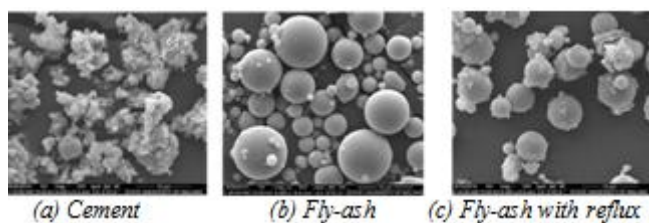


Figure 5: SEM Photo's (magnitude 8.000 times)

Scanning Electrone Microscopy (SEM) results with magnification 8000 times (see Figure-5) gained that fly-ash which has undergone the process of reflux will look granular synthesis-grain round-shaped and irregular spheres with a diameter of more refined (6,425 μm sized) compared with conventional fly-ash (9,956 μm sized) which means it has a greater specific surface area. Grains fineness on fly-ash also

resulting in reduced porosity on binder. It is closely related to reactivity of fly-ash is good because it makes it easier to harden when mixed with lime and water and ultimately improve the quality of binder on the concrete.

4. Conclusion

Characteristics of fly-ash have been synthesized by method of reflux has undergone a number of changes among other things ; fineness and consistency untill to 13.4% and 37% respectively, where that the potential has a large absorption rate compared with cement and ordinary fly-ash.

The chemical composition of fly-ash that have been synthesized by the method of reflux resulted increase of silica compounds (SiO_2) of 37,69% and has better reactivity with amorphous phase is formed untill 71,31%. Spherical granules with size more delicate of 6,425 μm will make the fly-ash synthesized by the method of reflux have better properties as cementitious compared with ordinary fly-ash has amorphous phase of 65,06% and more coarse grain size of 10,42 μm .

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